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January 12, 1987

Dr. C.C. Chao
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Ontario Waste Management Corporation
2 Bloor St. West
Toronto, Ontario
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Re: **Physical/Chemical Treatability Tests**

Dear Chih:

We are pleased to submit three (3) copies of our Final Report Entitled "Preliminary Physical/Chemical Treatability Tests".

This final report incorporates OWMC's comments where appropriate as discussed during our review meeting on December 11, 1986.

We trust this report meets your requirements. If you have any questions, please do not hesitate to call.

Sincerely,

R.P. Canning,
Manager, Process Engineering

RPC/mp
Encl.

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SUMMARY OF CONCLUSIONS AND RECOMMENDATIONS

The following points summarize the major conclusions and recommendations from this study:

- The results obtained from this study have been interpreted by comparing analytical data (before and after treatment) in terms of treatment efficiency or percentage removals. This is important because there is considerable analytical inaccuracy associated with analyzing the organic and inorganic contents of complex multi component waste mixtures. Similar inaccuracies have been reported by others (U.S.EPA, 1984). Although these inaccuracies exist and are unavoidable with currently available analytical methods and techniques, they tend to remain relatively consistant for different samples taken from the same waste matrix. Meaningful conclusions can therefore be drawn by comparing the results obtained at various stages of treatment for this particular waste matrix. However, caution should be exercised in comparing the results from this study with those obtained elsewhere on other waste matrices.
- The results have shown that physical/chemical treatment processes are capable of achieving effective removals of a wide range of volatile and non-volatile organic contaminants.
- For the low-level spiked waste, the combined physical/chemical treatment process resulted in a reduction of the concentrations of most contaminants measured in the treated residues by one to two orders of magnitude.

- Removal efficiencies were not as good for the high-level spiked waste compared to the low-level spiked waste particularly for the measured non-volatile organics which were shown to be reduced only one order of magnitude or less in the treated residues. The concentrations of measured volatiles on the other hand were reduced by two or three orders of magnitude for the high level spiked waste.
- A majority (> 90%) of the measured volatile organics were removed by the combined effects of emulsion breaking and air stripping.
- Emulsion breaking was effective for desorbing some of the measured non-volatile organics from the solid phase but had little or no effect on other non-volatiles. Compounds such as aniline, bis(2-chloroethyl)ether and lindane appeared to be effectively removed from the solid phase by the combined effects of heat and gravity sedimentation.
- Chemical oxidation did not appear to have a significant effect on the concentrations of any of the measured organics with the possible exception of 4-bromophenyl phenyl ether, benzene, 1,1-dichloroethylene and methylene chloride in the low level spiked waste and phenol in the high level spiked waste.
- In all of the samples measured, the TOC value of the waste mixture either stayed the same or increased after each stage of treatment. The reasons for this are not known. However, it is possible that chemical changes brought about by the treatment processes have caused changes in the measured

organics making them more readily detected by the TOC analysis. In view of this behaviour, it would appear that TOC is not a particularly good surrogate parameter for indicating the removal efficiency of groups of organics in a complex waste mixture.

- Coagulation/sedimentation appeared to have a negative effect on some of the measured non-volatile organics in the aqueous phase of the high level spiked waste. Although it is possible that some organics may become desorbed from the solids in the waste mixture as a result of the polymer addition in the coagulation process, additional investigations would be required to verify this possibility. Such investigations are beyond the scope of the present study.
- It is significant to note that although the measured non-volatiles were not very effectively removed by the treatment processes, particularly in the high level spiked waste, these compounds were not generally detectable in the filter cake washwater. This suggests that these organics are strongly adsorbed to the solids. Consequently, filter cake washing with water does not appear to be a particularly effective means of further reducing the concentrations of residual organics in the filtered solids.
- It is recommended that solidification and leaching tests be carried out on samples of the treated residues from this study to determine whether the residual organics which

remain in the treated and solidified filter cake are susceptible to leaching.

- It is recommended that future treatability testing experiments include provision for a trial run to establish analytical procedures (eg. dilution factors) and to check the suitability of the process conditions.
- It is recommended that future physical/chemical treatability and solidification testing be carried out on other "typical" inorganic wastes containing organics without the addition of spiked organics with a view to providing additional treatability and leach test data on a variety of different industrial wastes of the type which are likely to require treatment at the CWMC facility.

1.0 INTRODUCTION

OWMC's proposed physical/chemical treatment plant will be designed to treat wastes which are primarily inorganic in nature. The waste characterization studies which have been carried out to date by OWMC have shown that many industrial wastes which are classified as "inorganic" also contain varying concentrations of organic contaminants which must also be removed, destroyed and/or detoxified. The specific types and concentrations of organics likely to be present in these wastes are not known. Therefore, in addition to the primary role of the physical/chemical treatment system for removing and/or detoxifying inorganic contaminants, the design of the physical/chemical treatment system also incorporates a number of treatment steps such as emulsion breaking, gravity separation, dissolved air flotation, filtration, chemical oxidation, activated carbon adsorption, and reverse osmosis which are capable of removing immiscible and dissolved organic contaminants (OWMC[2]).

Unfortunately, regardless of the physical/chemical treatment processes used, it will not be possible to remove 100% of the immiscible and dissolved organics which are associated with the inorganic wastes being treated in the physical/chemical plant. A portion of the immiscible organics for example, will remain adsorbed to solid particles in the dewatered sludges. In addition, residual dissolved organics will also be present in the aqueous portion of these dewatered sludges which are comprised of approximately 65 - 70 wt% moisture (30 - 35% solids). Dissolved organics would also be present in the rejects from the reverse osmosis units and, to a lesser extent, in the evaporator residues.

Treated residues from the physical/chemical treatment processes which contain high concentrations of organics will be incinerated. Otherwise, these treated residues will be further treated in a solidification plant before being disposed of in an engineered landfill. Studies are currently being carried out by OWMC to assess the effectiveness of solidification processes for minimizing the leachability of different types of organic contaminants at different concentrations. The results of these tests will provide much needed data and information on the levels of organics which are capable of being effectively treated by solidification processes prior to disposal in the engineered landfill. In order to put the results of these solidification and leach testing studies into perspective, additional information is required on the effectiveness of physical/chemical treatment processes for removing organics and for minimizing the concentrations of organics in the treated residues. There is, at the present time, very little information available to date on the partitioning behaviour of organics between the solid and liquid phases in actual wastes which are subjected to physical/chemical treatment.

The purpose of this study is to provide the first step to collecting this information. The study focuses on using a single inorganic waste which contains some organics and is spiked with a range of additional organic contaminants of potential concern. Treatment was carried out using treatment processes of the type which have been included in the preliminary design of OWMC's physical/chemical treatment plant (OWMC^2).

Additional investigations of this type will be required to confirm the results obtained by this study using different wastes, and/or residues and a broader range of treatment process conditions.

2.0 OBJECTIVES

The objectives of this study can be summarized as follows:

- To obtain a preliminary indication of the fate of organic contaminants in a sample of inorganic waste subjected to physical/chemical treatment methods. In particular, to quantify the organic contaminants of concern which remain with the dewatered sludge after treatment.
- To utilize treatment methods which have been included in the preliminary design of the physical/chemical treatment plant.
- To obtain comparative results at two different treatment process conditions.

3.0 EXPERIMENTAL PROCEDURES

3.1 Selection of Waste Sample

A number of alternative industrial hazardous wastes were evaluated for their suitability for use in the test program. After consultation with OWMC, a partially treated slurry from the electroplating industry was chosen for testing. The criteria which were applied to the selection of the preferred waste samples for spiking and subsequent treatment were as follows:

- The waste contains 2% organics or less.
- The waste requires physical/chemical treatment (ie. contains heavy metals, reactive anions, etc.).
- The waste contains suspended solids (ie. is in the form of a slurry, cake or solid).
- The waste is likely to be available in quantities ranging from 500 to 1000 t/a or more.

Table 3-1 summarizes the chemical characteristics of the electroplating waste prior to spiking.

The analytical protocols used for the organic analyses are summarized in Appendix A. A summary of the minimum detection limits for each organic compound are presented in Table C-1, Appendix C.

3.2 Characterization of Waste Sample

A 45 gallon drum of waste sludge was obtained for the treatability experiments. Approximately 4 L of the waste was collected in a clean bottle for analysis. Since the waste had a high suspended solids content, it was mixed for about 5 minutes using a

Table 3.1

PROPERTIES OF UNTREATED WASTE
PRIOR TO SPIKING

Inorganics	Aqueous (mg/L)	Organics	Aqueous (ug/L)	Solid (ug/g)
Calcium	6.4	Benzene	3140	
Magnesium	0.44	Bis (2-ethylhexyl) phthalate	547	20
Sodium	690	Naphthalene		0.62
Potassium	980	Phenanthrene	8.5	
Aluminum	72	Phenol	<20	0.92
Barium	0.12	Trichloroethylene	11000	
Beryllium	< 0.0003			
Boron	4.1			
Cadmium	< 0.002			
Chromium	0.38			
Cobalt	<0.01			
Copper	21			
Iron	1.3			
Lead	< 0.04			
Manganese	0.01			
Molybdenum	< 0.02			
Nickel	0.07			
Phosphorous	5.1			
Silicon	1.1			
Silver	< 0.01			
Strontium	0.03			
Thallium	< 0.02			
Titanium	0.20			
Vanadium	< 0.02			
Zinc	0.24			
Zirconium	< 0.01			
Total CN	160			

Note: Parameters with unreported values were not analyzed.

drill mixer before sampling in order to ensure that a representative sample was collected.

The pH and total suspended solids of the waste were measured by following the procedures in Standard Methods. About 500 mL of the waste were filtered through a 0.45 u filter and the filtrate and filter cakes were analysed for their organic and inorganic concentrations. A detailed list of procedures for organic and inorganic analysis is given in Appendix A.

3.3 Spiking Solution Preparation

Twenty-two compounds, which represent a wide range of EPA organic priority pollutants, were selected to spike the waste sample. The spiking mixture was the same as that used by WTC in their solidification experiments on spiked synthetic waste. The types and concentrations of contaminants used in the experiments were chosen in consultation with OWMC. A majority of the organics were spiked at two concentration levels, 1,000 ug/g and 10 ug/g.

Since it was necessary to have approximately 8 L of sample for each of two treatment trains, the spiking solution recipe was prepared by calculating the amount of each individual compound required for a 20 L sample to ensure an adequate volume of waste was available for testing. These amounts are summarized in Table 3-2. The required mass of each chemical was weighed into a 500 mL glass bottle. The prepared spiking solution had the appearance of a homogeneous liquid.

The spiking solution (3.97 g) was weighed and added to 17 L of the waste sample in a 18 L glass jar for low level spiking. The

Table 3.2 Composition of Spiking Solution Mixture

CHEMICALS	WEIGHT ADDED TO SPIKING SOLUTION (g)
NON VOLATILES	
Acenaphthalene	2.1102
Aniline	20
Bis (2-chloroethyl) ether	20
4-Bromophenyl phenyl ether	20
Diethyl phthalate	20
1,2-Diphenylhydrazine	2.001
Phenol	20
p,p'-DDT	0.3515
Lindane	0.3545
Arochlor 1260 *	0.5396
VOLATILES	
Acrylonitrile	200
Benzene	20
1,1-Dichloroethylene	20
Dibromoethane	2
Methylene Chloride	20
1,1,2,2-Tetrachloroethane	20
1,1,1-Trichloroethane	20
Trichloroethylene	20
Toluene	20
Xylene	20

Note :

for low level spiking runs 3.97 g of this mixture was added to 17L of waste sample
for high level spiking runs 397 g of this mixture was added to 17L of waste sample
* 66% pure

sample was mixed overnight using a magnetic stirrer. A total of 397 g of the spiking solution was added to 17 L of the waste sample for high level spiking. This too was mixed overnight.

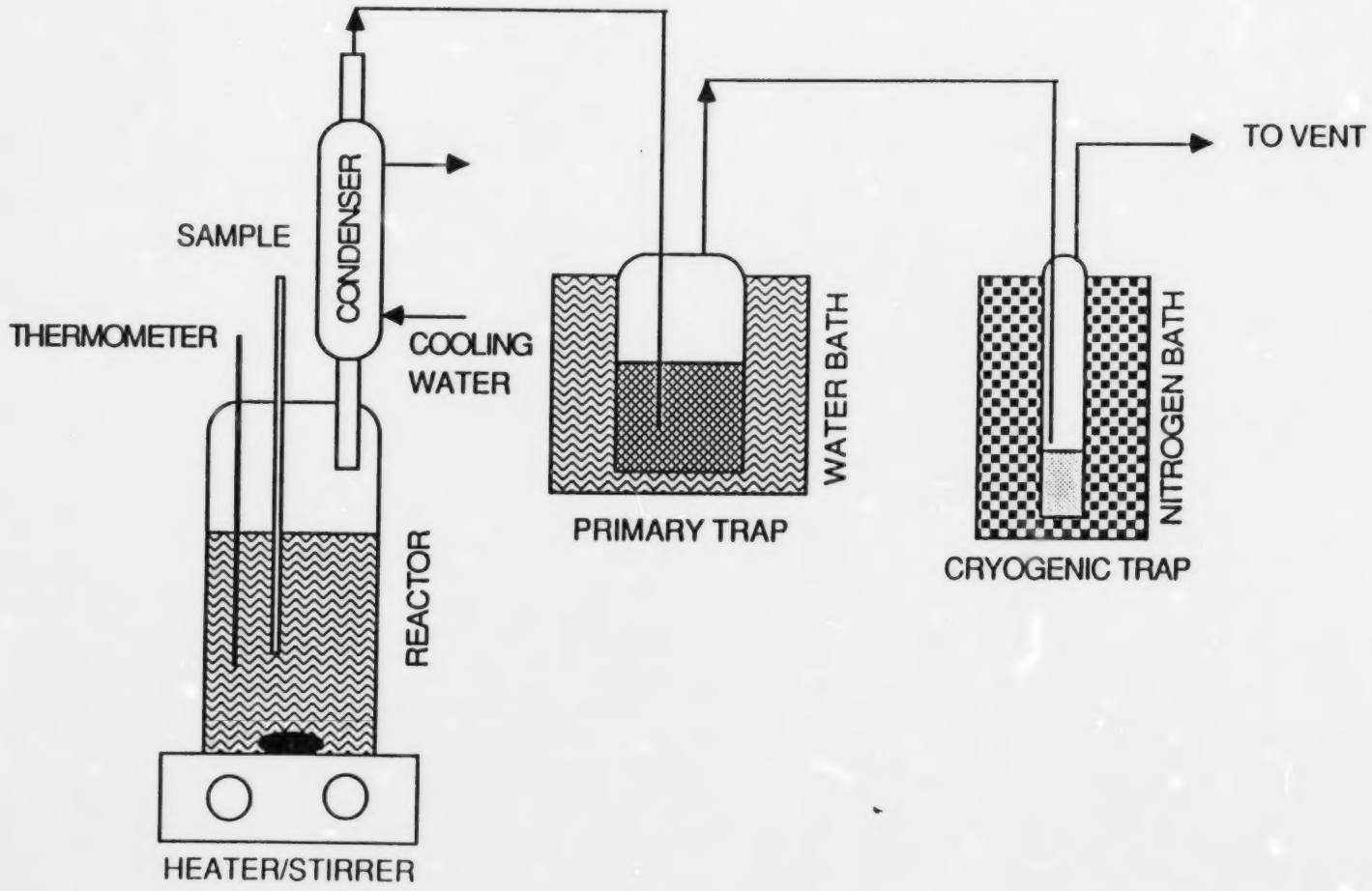
3.4 Waste Treatment Processes

3.4.1 Emulsion Breaking

The schematic flow sheet of the emulsion breaking process is given in Figure 3-1. A 10 L pyrex glass vessel containing 8 L of waste sample was placed on a heater/stirrer plate. The mouth of the glass vessel was sealed with a rubber stopper equipped with holes to accommodate a thermometer, sampling glass tube, an air sparger, and an adaptor for a condenser. The condenser was connected to the reaction vessel and cold tap water passed through it to recover water vapour and less volatile compounds. This ensured minimal loss of water over the 24 hour emulsion breaking period. The condenser vapour outlet was connected to a primary trap by teflon tubing. The primary trap consisted of a 250 mL Erlenmeyer flask containing approximately 100 mL of methanol. The flask was cooled in a water bath and sealed with a rubber stopper which had two holes to accommodate glass tubes; one as an inlet from the condenser vapour outlet and another as an outlet to a cryogenic trap. One end of the inlet tube was extended close to the bottom of the flask and completely submerged in the methanol solution. The outlet tube was positioned well above the methanol solution level to avoid potential entrainment of the solution. Teflon tubing was used to connect the outlet tube from the primary trap to the inlet of a cryogenic trap unit. The cryogenic units had individual flow control valves. One condenser was placed in

FIGURE 3-1

SCHEMATIC PROCESS FLOW SHEET FOR EMULSION BREAKING UNIT



liquid nitrogen solution in a thermos. The other control valves remained closed. The gas outlet from the organic trap condenser was open to the fume hood.

The following procedure was used for the emulsion breaking process:

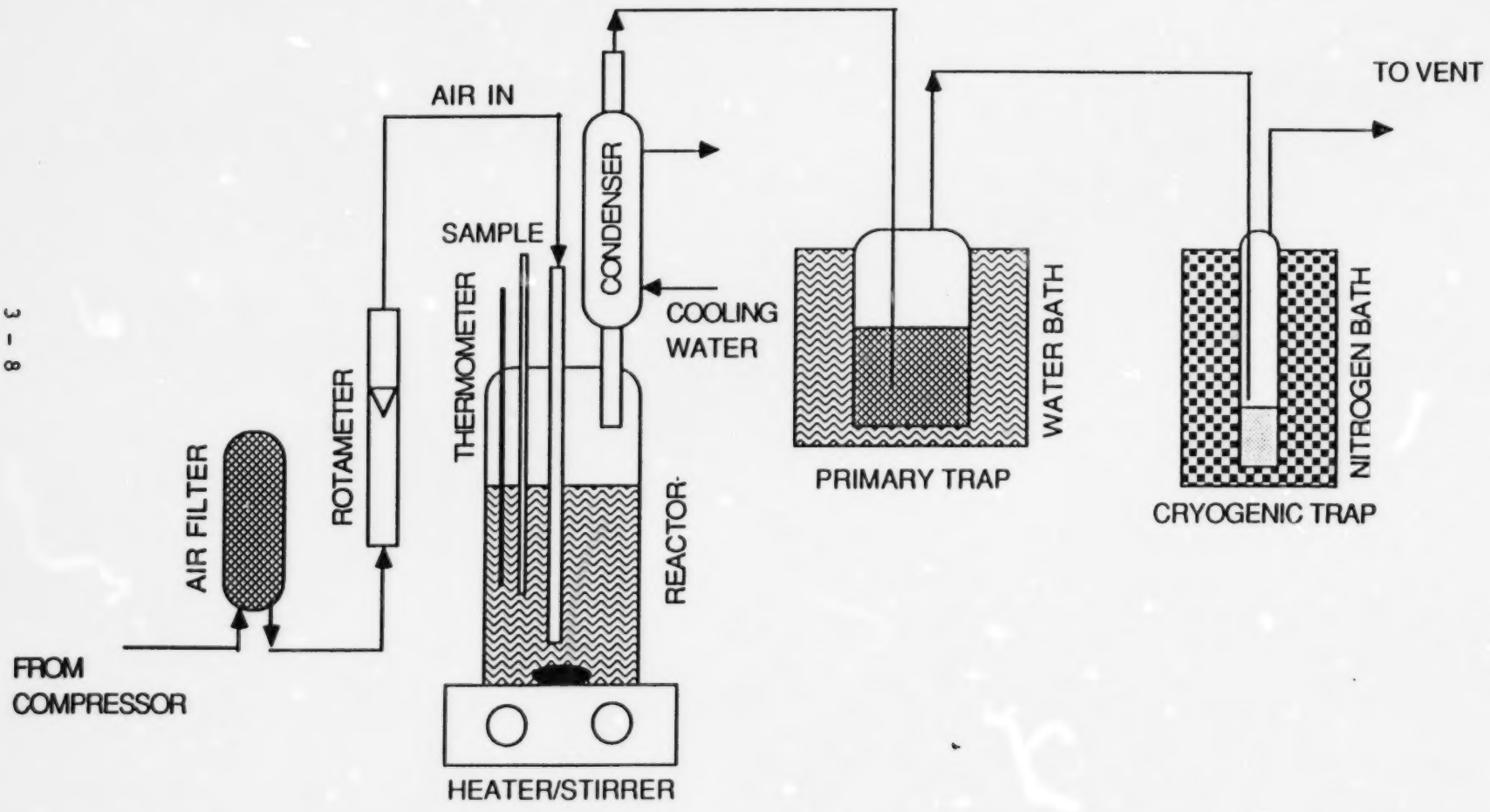
- Mixing and heating of the waste in the vessel was initiated by turning the stirrer and heater unit on.
- Cooling water flows to the condenser and water bath were also started.
- The temperature of the waste sample was raised to 80°C and given mild agitation for 2 hours.
- The magnetic stirrer was turned off after two hours and the temperature of the sample was maintained at 80°C for an additional 20 hours to complete the emulsion breaking step.
- About 250 mL of the waste sample and 100 mL of methanol in the primary trap were collected for analysis at the end of the process.
- The cryogenic condenser was removed and stored in a freezer prior to analysis.

3.4.2 Air Stripping

Air stripping was carried out in the same apparatus used for the emulsion breaking step with only slight modifications as shown in Figure 3-2. Air flow was introduced through a sparger which was positioned close to the bottom of the glass vessel. An air compressor with a pressure regulator was used to generate compressed air. The air flow rate was measured by a precalibrated rotameter. Two

FIGURE 3-2

SCHEMATIC PROCESS FLOW SHEET FOR AIR STRIPPING UNIT



cryogenic condensers were used during the air stripping process to ensure that all of the organic laden vapours were collected for analysis.

The following procedure was used for the air stripping step:

- After the emulsion breaking step, the primary trap was replaced and two new cryogenic condensers were installed and replenished with liquid nitrogen.
- The temperature of the waste sample was maintained at 80°C.
- The air flow rate through the sparger was maintained at a flow rate of 2 L/min for 2 hours.
- The cooling water flow rates for the condenser and the water bath were maintained at the same levels as for the emulsion breaking step.
- Samples of waste methanol from the primary trap and the contents of the two cryogenic traps were collected for analysis at the end the of two hours of air stripping.

3.4.3 Chemical Oxidation

The glass vessel was moved from the emulsion breaking/air stripping apparatus to a magnetic stirrer and cooled to ambient temperature after air stripping. A pH electrode and an oxidation reduction potential electrode were placed in the vessel.

The following procedure was used for the chemical oxidation step.

- The pH of the waste sample was adjusted to 10 by either adding 10% by weight lime slurry or 5N-H_2SO_4.

- The redox potential of the solution was measured and 12% NaOCl solution was added to maintain a redox potential of approximately 350 mV.
- When the pH and redox potential stabilized to pH 10 and 350 mV respectively, the second stage oxidation was begun.
- The pH of the solution was adjusted to 8 and additional NaOCl solution was added to maintain a redox potential of 600 mV until the pH and redox potential stabilized.
- About a 250 mL sample of the waste was collected for analysis at the end of chemical oxidation process.

3.4.4 Neutralization and Precipitation

After chemical oxidation, the pH of the solution was adjusted to 9.5 by adding 10% $Ca(OH)_2$ slurry while the solution was being mixed. About 10 minutes of reaction time was allowed to ensure a complete neutralization.

3.4.5 Coagulation

The separation of precipitated products was done by a coagulation process. A strongly anionic coagulant (Purifloc A23) was added to the solution at a concentration of 20 ppm under a rapid mixing condition. The rapid mixing continued for one minute followed by four minutes of slow mixing. At the end of the slow mixing period, the solution was allowed to settle for approximately one hour. The supernatant of the settled solution was carefully withdrawn for analysis using a glass tube.

3.4.6 Filtration

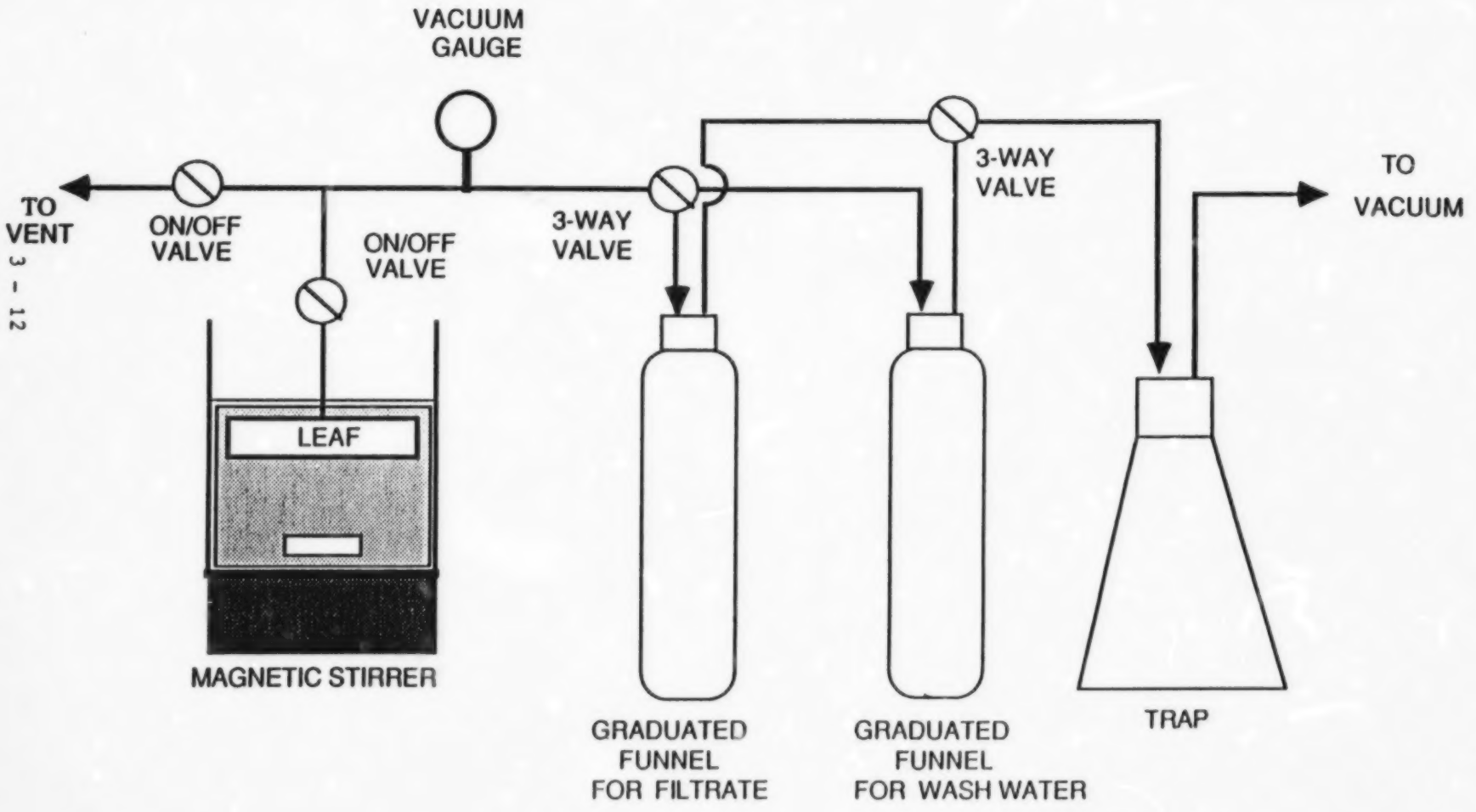
Filtration of the settled sludge sample after coagulation was carried out in the vacuum leaf filter test system. This apparatus is shown schematically in Figure 3-3. A Dorr-Oliver leaf filter unit having a 81 cm^2 of filtration area was used in this experiment. A nylon filter cloth (National Filter Media N1001) with medium porosity was fastened to the face of the filter using a hose clamp. A vacuum was generated by an aspirator and controlled by adjusting water flow rate. The vacuum line from the leaf filter was connected to a graduated funnel via stainless steel tubing and tygon tubing. The outlet of the funnel was connected to a holding flask whose outlet was connected to the aspirator. A vacuum gauge was placed between the leaf filter and the graduated funnel. The filtrate was collected in the graduated funnel during a filtration run. A magnetic stirrer was used to gently mix the slurry sample during filtration.

The following procedure was used for the filtration.

- About 1500 mL of sludge were placed in a glass vessel.
- The water to the aspirator was turned on and a vacuum of about 20 mm.Hg was established.
- The filter leaf was submerged in the sludge and the valve to the filter leaf opened.
- The volume of filtrate and pressure were recorded at 20 second intervals for 7 to 10 minutes.
- The filter leaf was removed from the sludge and the filter cake allowed to dry for approximately one minute.
- The 3-way valves were adjusted to collect wash water.

FIGURE 3-3

SCHEMATIC FLOW SHEET OF THE VACUUM LEAF TEST UNIT



- The filter cake was submerged in about 1500 mL of deionized water.
- The filter cake was washed until about 50 mL of wash water were collected.
- The leaf filter was removed from the deionized water and the filter cake was dried.

4.0 RESULTS AND DISCUSSION

4.1 Status of Data

Tables 4-1 to 4-3 show the actual quantities of each of the non-volatile organic contaminants which were spiked into the untreated waste. These values are compared to the quantities of each measured organic which can be accounted for by analysis of each spiked sample. The results for train 2 with the low level spiking solution have not been included because these data are identical to those presented in Table 4-1 for train 1. In this case, the spiked sample was split prior to treatment in each train. For the high level spiked case, the analyses of the spiked feeds to trains 1 and 2 were slightly different; therefore, both sets of results are presented (Tables 4-2 and 4-3).

Although similar data on the measured volatile organics have not been included in Tables 4-1 to 4-3 because volatiles were not analyzed in both the aqueous and solid phases of each waste sample, the data given in Tables 4-1 to 4-3 clearly demonstrate the degree of inaccuracy which is unavoidable in carrying out analyses of complex multi component waste mixtures. Although the "Percentage Detected" values range from less than 10% to over 600%, a majority of the results fall into the ranges which are generally accepted by the U.S.EPA (ref: U.S.EPA, 1984) for analyses of this type. Analytical results were not obtained for 1,2-diphenylhydrazine. This compound appeared to be degraded in the presence of the other organics in the mixture. This behaviour could account for the apparently anomalous results obtained for 1,2-diphenylhydrazine (see Section B.2, Appendix B).

Table 4.1 Status of Data- Train I with Low Level Spike

Measured nonvolatile organics	Quantity spiked into waste sample (mg)	Quantity Detected in Untreated Sample		% detected vs Quantity Spiked	% detected approved by EPA*
		liquid (mg)	solid (mg)		
Acenaphthalene	7.91	0.00	6.90	87.3%	47-145
Aniline	74.95	15.75	2.89	24.9%	NS
Bis (2-chloroethyl) ether	74.95	30.00	1.95	42.6%	12-158
4-Bromophenyl phenyl ether	74.95	0.00	194.53	259.6%	53-127
Diethyl phthalate	74.95	0.00	4.52	6.0%	ND-114
1,2-Diphenylhydrazine	0.75	0.00	20.08 **	NA	NS
Phenol	74.95	15.00	1.44	21.9%	5-112
p,p'-DDT	1.32	0.00	3.18	241.1%	ND-203
Lindane	1.33	0.00	8.72	657.0%	NS
Arochlor 1260	1.33	0.00	0.43	32.0%	ND-164

Note:

ND = Not detected

NS = Not Specified NA = Not Applicable

* Source : U.S. EPA

** : Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)

Table 4.2 Status of Data- Train I with High Level Spike

Measured nonvolatile organics	Quantity spiked into waste sample (mg)	Quantity Detected in Untreated Sample		% detected vs Quantity Spiked	% detected approved by EPA*
		liquid (mg)	solid (mg)		
Acenaphthalene	527.19	0.00	313.76	59.5%	47-145
Aniline	4996.36	900.00	163.16	21.3%	NS
Bis (2-chloroethyl) ether	4996.36	950.00	389.07	26.8%	12-158
4-Bromophenyl phenyl ether	4996.36	0.00	3012.12	60.3%	53-127
Diethyl phthalate	4996.36	0.85	2384.60	47.7%	ND-114
1,2-Diphenylhydrazine	49.98	0.00	460.19 **	NA	NS
Phenol	4996.36	220.00	121.32	6.8%	5-112
p,p'-DDT	87.81	0.00	108.77	123.9%	ND-203
Lindane	88.51	0.00	90.36	102.1%	NS
Arochlor 1260	88.97	0.00	10.84	12.2%	ND-164

Note:
ND = Not detected
NS = Not Specified NA = Not Applicable
* Source : U.S. EPA
** : Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)

Table 4.3 Status of Data- Train II with High Level Spike

Measured nonvolatile organics	Quantity spiked into waste sample (mg)	Quantity Detected in Untreated Sample		% detected vs Quantity Spiked	% detected approved by EPA*
		liquid (mg)	solid (mg)		
Acenaphthalene	527.19	0.00	305.40	57.9%	47-145
Aniline	4996.36	2750.00	129.69	57.6%	NS
Bis (2-chloroethyl) ether	4996.36	1450.00	221.73	33.5%	12-158
4-Bromophenyl phenyl ether	4996.36	0.00	2593.77	51.9%	53-127
Diethyl phthalate	4996.36	0.00	2426.43	48.6%	ND-114
1,2-Diphenylhydrazine	49.98	0.00	460.19 **	NA	NS
Phenol	4996.36	340.00	92.04	8.6%	5-112
p,p'-DDT	87.81	0.00	11.92	13.6%	ND-203
Lindane	88.51	0.00	95.80	108.2%	NS
Arochlor 1260	88.97	0.00	16.06	18.1%	ND-164

Note:
ND = Not detected
NS = Not Specified NA = Not Applicable
* Source : U.S. EPA
** : Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)

It is significant to note that the results which were obtained on the two similar high level spiked waste samples (Tables 4-2 and 4-3) produced very similar values in terms of "Percentages Detected". The only exception to this was DDT although even these results were within the percentage detected range which is generally accepted by the EPA.

The major conclusion which can be drawn from the results shown in Tables 4-1 to 4-3 is that the actual values reported for the measured organics are subject to considerable analytical inaccuracy. However, since these inaccuracies can be expected to apply equally to the analyses performed both before and after treatment, a comparison of these analytical results, expressed in terms of treatment efficiency or percentage removals, still provides a valid and meaningful basis for interpreting these data.

4.2 Results of Trial Run

A trial run of the treatment process was carried out with a low level spiked waste in order to determine the proper operating conditions for each unit operation and to establish appropriate procedures and protocols for analyzing the required samples of air, water, and solids. A number of difficulties were experienced in analyzing some samples particularly samples from the methanol and cryogenic traps which contained much higher concentrations of measured volatile organics than originally anticipated. These aspects are discussed further in Appendix B.

Table 4-4 summarizes the operating conditions used for the trial run with the low level spiked sample.

Table 4.4 PROCESS OPERATING CONDITIONS FOR THE TRIAL RUN

PROCESS	OPERATING CONDITIONS			CHEMICALS	OBSERVATIONS
	PH	TEMP.(C)	CONTACT TIME		
EMULSION BREAKING		80+5	2 hr. mixing 17.5 hr. separating		Sample Volume = 8 L It took 2 hrs. to heat up to 80 'C
AIR STRIPPING		80+5	2 hr. mixing	Air Flow 2 L/min	It took 2.5 hrs. to cool to ambient temperature
CHEMICAL OXIDATION First Stage Second Stage	initial 10.6 final 10.5 final 8.48	20+1	1 hr. mixing 21 hr. mixing	8.51 g. NaOCl 14.31g. NaOCl	 Required 175 mL 1N H_2SO_4 to adjust pH to 8.5
NEUTRALIZATION/ PRECIPITATION	initial 8.47 final 9.5	20+1	10 min. mixing	17 ml 10% wt $Ca(OH)_2$ 500 ml 0.1N $Ca(OH)_2$	
COAGULATION		20+1	5 min. mixing 2 hr. settling	20 mg/L 0.5% Purifloc	Supernatant 2 L
FILTRATION		20+1	Filtering 3 min. Drying = 1 min. Washing = 3.0 min. Drying = 1.0 min.	Vacuum ~ 20 mmHg	Collected - 690 mL Filtrate - 200 mL wash water Filter Cake - 75 % moisture

The trial run revealed a number of operational factors which were taken into consideration in the subsequent experimental runs.

- It was determined from the trial run that for the low level spiked waste, a relatively large volume (approximately 500 mL) of sample would be required for each set of analyses in order to obtain sufficient contaminant for accurate analysis.
- The waste required two to three hours to heat up to 80°C at the start of the emulsion breaking step and an additional two to three hours to cool down after air stripping. Consequently, the scheduling of the treatment sequences was adjusted to accommodate these long heating and cooling times.
- Large volumes of acids and alkalis were required to adjust the solution pH using dilute reagents. Consequently, concentrated reagents were used for the actual test runs to minimize the reagent volumes required.
- Coagulant dosages ranging from 1 to 50 ug/g were tested during the trial run. The optimum dosage for Purifloc A23 was determined to be in the range of 10 to 20 ug/g.
- The trial runs showed that a significant quantity of methanol would be carried over from the primary trap and captured in the cryogenic trap during air stripping. Consequently, two cryogenic traps were used in parallel during air stripping in order to minimize the volume of air flow through each trap.

- The settling rate of the sludge in the coagulation step was extremely slow and consequently additional care had to be taken in separating the supernatant after settling to avoid disturbing the settled sludge.
- It was difficult to maintain a constant vacuum level during the filtration step. This was unavoidable due to the build up of solids on the filter leaf.
- Because of the high suspended solids content of the waste, the magnetic stirrer provided only mild agitation. Future experiments of this nature will require that some provision be made for more vigorous agitation of the waste to ensure more rapid mixing during the reaction sequence.

Tables 4-5 and 4-6 present the analytical results from the trial run. It is important to emphasize that the trial run involved making adjustments to both the test procedures and the analytical procedures. For this reason, these results are considered to be less accurate than those obtained from the subsequent test runs. Consequently, the results of the trial run have not been interpreted in detail. In general terms however, the following observations can be made from these data:

- Compounds which have low water solublities and high sediment/water partitioning coefficients (eg. acenaphthalene, 4-bromophenyl phenyl ether, lindane and PCB) were generally not detectable in the liquid phase of the sample.

Table 4.5 Analytical Results for Trial Run

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	(mg/L)	filtrate (mg/L)	wash water (mg/L)	filter cake (ug/g)
Non-volatiles										
Acenaphthalene	ND	0.37	ND	0.41	ND	0.12	ND	ND	0.004	16
Aniline	4	0.063	4.3	0.019	2.7	0.023	1	1.3		ND
Bis (2-chloroethyl) ether	10.2	ND	4	ND	3.3	ND	2.2	1.8	2.1	ND
4-Bromophenyl phenyl ether	ND	4	0.019	6.1	ND	2.4	0.01	0.02	0.057	520
Diethyl phthalate	0.26	0.17	0.15	0.32	0.026	0.14	ND	ND	ND	ND
1,2-Diphenylhydrazine*	ND	ND	ND	0.78	ND	0.22	0.1	0.05	0.098	31
Phenol	1.9	ND	2.3	0.009	2.3	ND	1	1	0.62	ND
p,p'-DDT	0.05	2.1	0.05	2.69	ND	1.09	ND	ND	ND	6.9
Lindane	ND	9.4	ND	ND	ND	ND	ND	ND	ND	ND
Arochlor 1260	ND	0.4	ND	NA	***	0.53	ND	ND	ND	1.21
Volatiles										
Acrylonitrile	ND		ND		ND		ND	ND	ND	
Benzene	2.7		1.1		ND		ND	0.01	0.005	
1,1-Dichloroethylene	180		ND		ND		ND	ND	ND	
Dibromoethane	ND		ND		ND		ND	ND	ND	
Methylene Chloride	13		ND		ND		0.22	0.06	ND	
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND	ND	ND	
1,1,1-Trichloroethane	10		ND		ND		ND	ND	ND	
Trichloroethylene	190		31		ND		0.59	0.17	0.038	
Toluene	9.4		2.7		ND		0.048	0.04	0.003	
Xylene	1.8		0.4		ND		0.021	0.01	0.002	

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix-B)

** Supernatant After Chemical oxidation, Neutralization/Precipatation, Coagulation and Sedimentation

ND: Not Detected at The Minimum Detection Limits NA : Not Applicable

*** Result Not Available

Table 4.6 Treatment Efficiency Results for Trial Run

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid	solid	liquid	solid		filtrate	wash water	filter cake
Non-volatiles										
Acenaphthalene	ND	0.37	ND	-11%	ND	68%	ND	ND	NA	-4224%
Aniline	4	0.063	-8%	70%	33%	63%	75%	68%	NA	100%
Bis (2-chloroethyl) ether	10.2	ND	61%	100%	68%	100%	78%	82%	NA	100%
4-Bromophenyl phenyl ether	ND	4	ND	-53%	ND	40%	ND	ND	NA	-12900%
Diethyl phthalate	0.26	0.17	42%	-88%	90%	18%	100%	100%	NA	100%
1,2-Diphenylhydrazine*	ND	ND	ND	ND	ND	ND	-100%	0%	NA	-3000%
Phenol	1.9	ND	-21%	100%	-21%	100%	47%	47%	NA	100%
p,p'-DDT	0.05	2.1	0%	-28%	100%	48%	100%	100%	NA	-229%
Lindane	ND	9.4	ND	100%	ND	100%	ND	ND	NA	100%
Arochlor 1260	ND	0.4	ND	100%	***	-33%	ND	ND	NA	-203%
Volatiles										
Acrylonitrile	ND		ND		ND		ND	ND	NA	
Benzene	2.7		59%		100%		100%	100%	NA	
1,1-Dichloroethylene	180		100%		100%		100%	100%	NA	
Dibromoethane	ND		ND		ND		ND	ND	NA	
Methylene Chloride	13		100%		100%		98%	100%	NA	
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND	ND	NA	
1,1,1-Trichloroethane	10		100%		100%		100%	100%	NA	
Trichloroethylene	190		84%		100%		100%	100%	NA	
Toluene	9.4		71%		100%		99%	100%	NA	
Xylene	1.8		78%		100%		99%	99%	NA	

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)

** Supernatant After Chemical oxidation, Neutralization/Precipatation, Coagulation and Sedimentation

ND: Not Detected at The Minimum Detection Limits NA : Not Applicable

*** Result Not Available

- Percentage values are cumulative and negative values indicate increasing concentrations

- The results shown in Table 4-6 indicate that essentially all of the measured volatiles had been removed from the aqueous phase of the waste by the combined emulsion breaking and air stripping steps. The filter cake washwater did not contain any detectable concentrations of these volatile compounds. Analyses of volatile compounds in the final filter cake were not performed because such results would not be particularly meaningful in view of the vacuum filtration and air drying steps required to produce the final filter cake.
- The results shown in Table 4-6 also indicate in general, that very high removal efficiencies are achievable for a majority of the measured organics.

4.3 Treatment of Low Level Spiked Waste

4.3.1 Treatment Process Train 1

Table 4-7 summarizes the operating conditions for each unit operation in process train 1.

The emulsion breaking step produced a very thin layer of dark scum on the surface of the sample. This material was left in the sample for the subsequent treatment steps. The chemical oxidation step did not produce any observable change in the appearance of the samples. In the coagulation step, a clear supernatant was obtained after approximately two hours of settling. The Purifloc A23 produced small slow settling flocs even at its optimum dosage. A total of 37.2 g of filter cake was recovered after vacuum filtration of the sludge. The cake was easily removed from the filter cloth and had a solids content of 25 wt%. A filtration rate of 0.29 mL/min. cm^2 was

Table 4.7 PROCESS OPERATING CONDITIONS FOR TRAIN 1 LOW LEVEL SPIKE

PROCESS	OPERATING CONDITIONS			CHEMICALS	OBSERVATIONS
	PH	TEMP.(C)	CONTACT TIME		
EMULSION BREAKING		80+5	2 hr. mixing 18 hr. separating		It took 2 hr. to heat up to 80 'C ~ 1/4 L scum on top layer ~ 1 L green aqueous middle layer ~ 6 L of sludge on bottom layer It took 3 hrs. to cool to ambient temperature
CHEMICAL OXIDATION First Stage	initial 10.6 final 10.5	20+1	2 hr. mixing	7.97 g. NaOCl	Required 6 mL 5N H2SO4 to adjust pH to 8.5.
Second Stage	final 8.5		21 hr. mixing	13.42 g. NaOCl	Required 50 mL 5N H2SO4 +12 mL 10 % wt. Ca(OH)2 to adjust pH to 10.5
NEUTRALIZATION/ PRECIPITATION	initial 9.0 final 9.5	20+1	30 min. mixing	19 mL 10% wt Ca(OH)2	
COAGULATION		20+1	5 min. mixing 2 hr. settling	20 mg/L 0.5% with Puriflo	Supernatant = 2 L
FILTRATION		20+1	Filtering 7.5 min. Drying 1 min. Washing 3 min. Drying 2 min.	Vacuum ~ 20 mmHg	Collected - 180 mL Filtrate - 90 mL Wash water Filter Cake - 37.2 g - 3.5 mm thick - 75% moisture

achieved during the filtration cycle, during which time the vacuum increased from 10 mm Hg to 25 mm Hg as the filter cake became thicker.

Tables 4-8 and 4-9 summarize the analytical results for train 1 with the low level spiked waste.

4.3.2 Treatment Process Train 2

Table 4-10 summarizes the operating conditions for each unit operation in process train 2 and tables 4-11 and 4-12 summarize the analytical results for train 2 with the low level spiked waste.

4.3.3 Discussion of Results from Treatment of Low Level Spiked Waste

The following observations and conclusions have been drawn from the results of treating the low level spiked waste.

- A majority of the measured non-volatile organics were not detectable in the aqueous phase of the feed samples (Tables 4-8 and 4-11). This is expected due to the high solid/liquid partitioning coefficients and the low water solubilities of these compounds. (see Table C-1, Appendix C)
- Compounds such as aniline, bis-2-chloroethyl ether and phenol tended to partition about equally between the solid and liquid phases. This is due to their relatively high water solubilities. (see Table C-1, Appendix C)

Combined Treatment Processes

- A majority of the measured non-volatile organics were very effectively removed from the solid phase by the combined

Table 4.8 Analytical Results for Train I- Low Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	(mg/L)	Filtrate (mg/L)	wash water (mg/L)	filter cake (ug/g)
Non-volatiles										
Acenaphthalene	ND	11	ND	10	ND	9.8	ND	0.001	ND	ND
Aniline	2.1	4.6	2.4	ND	2.2	1.7	2.7	1.2	0.15	ND
Bis (2-chloroethyl) ether	4	3.1	3	ND	3	ND	5.3	2.1	0.34	ND
4-Bromophenyl phenyl ether	ND	310	ND	220	ND	ND	ND	0.079	0.002	1.6
Diethyl phthalate	ND	7.2	ND	ND	0.01	ND	ND	0.049	ND	ND
1,2-Diphenylhydrazine*	ND	32	ND	17	ND	19	0.024	ND	ND	ND
Phenol	2	2.3	0.93	ND	1.5	ND	1.9	0.66	0.062	ND
p,p'-DDT	ND	5.06	ND	1.06	ND	1.16	ND	ND	ND	0.34
Lindane	ND	13.9	ND	0.896	ND	0.76	ND	ND	ND	0.43
Arochlor 1260	ND	0.681	ND	0.54	ND	0.72	ND	ND	ND	0.42
Volatiles										
Acrylonitrile	11		ND		ND		ND	ND	ND	
Benzene	2.8		2.6		0.52		0.15	ND	ND	
1,1-Dichloroethylene	6.7		8.1		1.1		ND	ND	ND	
Dibromoethane	2		ND		ND		ND	ND	ND	
Methylene Chloride	1.2		1.1		0.17		ND	ND	ND	
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND	ND	ND	
1,1,1-Trichloroethane	2.8		0.84		0.19		ND	ND	ND	
Trichloroethylene	44		58		27		17	1.3	ND	
Toluene	2.1		2.2		0.75		0.22	0.39	ND	
Xylene	0.21		0.62		0.24		ND	ND	ND	
Gross Parameter										
TOC	495		660		440			495		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)
ND: Not Detected at The Minimum Detection Limits NA : Not Applicable
** Supernatant After Coagulation/Sedimentation

Table 4.9 Treatment Efficiency Results for Train I - Low Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid	solid	liquid	solid		Filtrate	wash water	filter cake
Non-volatiles										
Acenaphthalene	ND	11	ND	9%	ND	11%	ND	ND	NA	100%
Aniline	2.1	4.6	-14%	100%	-5%	63%	-29%	43%	NA	100%
Bis (2-chloroethyl) ether	4	3.1	25%	100%	25%	100%	-33%	48%	NA	100%
4-Bromophenyl phenyl ether	ND	310	ND	29%	ND	100%	ND	-58%	NA	99%
Diethyl phthalate	ND	7.2	ND	100%	ND	100%	ND	ND	NA	100%
1,2-Diphenylhydrazine*	ND	32	ND	47%	ND	41%	ND	ND	NA	100%
Phenol	2	2.3	54%	100%	25%	100%	5%	67%	NA	100%
p,p'-DDT	ND	5.06	ND	79%	ND	77%	ND	ND	NA	93%
Lindane	ND	13.9	ND	94%	ND	95%	ND	ND	NA	97%
Arochlor 1260	ND	0.681	ND	21%	ND	-6%	ND	ND	NA	38%
Volatiles										
Acrylonitrile	11		100%		100%		100%	100%	NA	
Benzene	2.8		7%		81%		95%	100%	NA	
1,1-Dichloroethylene	6.7		-21%		84%		100%	100%	NA	
Dibromoethane	2		100%		100%		100%	100%	NA	
Methylene Chloride	1.2		8%		86%		100%	100%	NA	
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND	ND	NA	
1,1,1-Trichloroethane	2.8		70%		93%		100%	100%	NA	
Trichloroethylene	44		-32%		39%		61%	97%	NA	
Toluene	2.1		-5%		64%		90%	81%	NA	
Xylene	0.21		-195%		-14%		100%	100%	NA	
Gross Parameter										
TOC	495		-33%		11%			0%		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)

ND: Not Detected at The Minimum Detection Limits NA : Not Applicable

- Percentage values are cumulative and negative values indicate increasing concentrations

** Supernatant After Coagulation/Sedimentation

Table 4.10 PROCESS OPERATING CONDITIONS FOR TRAIN 2 -LOW LEVEL SPIKE

PROCESS	OPERATING CONDITIONS			CHEMICALS	OBSERVATIONS
	PH	TEMP.(C)	CONTACT TIME		
EMULSION BREAKING		80+5	2 hr. mixing 18 hr. separating		Sample Volume = 7.5 L It took 2 hr. to heat up to 80 'C
AIR STRIPPING		80+5	2 hr. mixing	Air Flow 2 L/min	It took 2.5 hr. to cool to ambient temperature
CHEMICAL OXIDATION First Stage Second Stage	initial 10.6 final 10.5 final 8.5	20+1	 21 hr. mixing	7.97 g. NaOCl 12.02g. NaOCl	Required 3 mL 5N H2SO4 to adjust pH to 10.5 Required 34.5 mL 5N H2SO4 to adjust pH to 8.5
NEUTRALIZATION/ PRECIPITATION	initial 8.47 final 9.5	20+1	30 min. mixing	15 mL 10% wt Ca(OH)2	
COAGULATION		20+1	5 min. mixing 2 hr. settling	20 mg/L 0.5% Purifloc	Supernatant 2 L
FILTRATION		20+1	Filtering 10 min. Drying = 1.5 min. Washing = 8 min. Drying = 2.0 min.	Vacuum ~ 20 mmHg	Collected - 150 mL Filtrate - 50 mL wash water Filter Cake - 68.9 g -5.6 mm thickness -76 % moisture

Table 4.11 Analytical Results for Train II- Low Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		After Chemical Oxida		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	(mg/L)	Filtrate (mg/L)	wash water (mg/L)	filter cake (ug/g)
Non-volatiles												
Acenaphthalene	ND	11	ND	13	ND	ND	ND	0.18	ND	ND	ND	8.7
Aniline	2.1	4.6	1.9	1.1	4.2	ND	3.8	ND	2.6	4.3	0.35	ND
Bis (2-chloroethyl) ether	4	3.1	0.7	ND	1.8	ND	2.3	ND	2.5	5.6	0.43	ND
4-Bromophenyl phenyl ether	ND	310	ND	200	ND	59	ND	2.6	ND	ND	ND	120
Diethyl phthalate	ND	7.2	ND	14	ND	ND	ND	ND	ND	ND	ND	16
1,2-Diphenylhydrazine*	ND	32	ND	17	ND	1.4	ND	0.075	0.1	ND	ND	ND
Phenol	2	2.3	1.3	0.5	1.5	ND	1.4	ND	2.3	1.9	0.14	ND
p,p'-DDT	ND	5.06	ND	0.38	ND	0.17	ND	0.59	ND	ND	ND	0.5
Lindane	ND	13.9	ND	0.21	ND	ND	ND	ND	ND	ND	ND	ND
Arochlor 1260	ND	0.681	ND	0.39	ND	0.22	ND	0.63	ND	ND	ND	0.47
Volatiles												
Acrylonitrile	11		ND		ND		ND		ND	ND	ND	
Benzene	2.8		1.4		0.09		0.04		ND	0.3	ND	
1,1-Dichloroethylene	6.7		2.7		0.02		ND		ND	0.26	ND	
Dibromoethane	2		ND		ND		ND		ND	ND	ND	
Methylene Chloride	1.2		0.59		ND		0.03		ND	ND	ND	
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND		ND	ND	ND	
1,1,1-Trichloroethane	2.8		0.08		ND		ND		ND	0.04	ND	
Trichloroethylene	44		38		1.4		0.87		0.4	9.2	ND	
Toluene	2.1		1.2		0.06		0.05		0.05	0.34	ND	
Xylene	0.21		0.39		0.01		0		0.01	0.1	ND	
Gross Parameter												
TOC	495		517		698		550			506		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)
ND: Not Detected at The Minimum Detection Limits NA : Not Applicable
** Supernatant After Coagulation/Sedimentation

Table 4.12 Treatment Efficiency Results for Train II -Low Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		After Chemical Oxida		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid	solid	liquid	solid	liquid	solid		Filtrate	wash water	filter cake
Non-volatiles												
Acenaphthalene	ND	11	ND	-18%	ND	100%	ND	98%	ND	100%	NA	21%
Aniline	2.1	4.6	10%	76%	-100%	100%	-81%	100%	-24%	-105%	NA	100%
Bis (2-chloroethyl) ether	4	3.1	83%	100%	55%	100%	43%	100%	38%	-40%	NA	100%
4-Bromophenyl phenyl ether	ND	310	ND	35%	ND	81%	ND	99%	ND	ND	NA	61%
Diethyl phthalate	ND	7.2	ND	-94%	ND	100%	ND	100%	ND	ND	NA	-122%
1,2-Diphenylhydrazine*	ND	32	ND	47%	ND	96%	ND	100%	-100%	ND	NA	100%
Phenol	2	2.3	35%	78%	25%	100%	30%	100%	-15%	5%	NA	100%
p,p'-DDT	ND	5.06	ND	92%	ND	97%	ND	88%	ND	ND	NA	90%
Lindane	ND	13.9	ND	98%	ND	100%	ND	100%	ND	ND	NA	100%
Arochlor 1260	ND	0.681	ND	43%	ND	68%	ND	7%	ND	ND	NA	31%
Volatiles												
Acrylonitrile	11		100%		100%		100%		100%	100%	NA	
Benzene	2.8		50%		97%		99%		100%	89%	NA	
1,1-Dichloroethylene	6.7		60%		100%		100%		100%	96%	NA	
Dibromoethane	2		100%		100%		100%		100%	100%	NA	
Methylene Chloride	1.2		51%		100%		98%		100%	100%	NA	
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND		ND	ND	NA	
1,1,1-Trichloroethane	2.8		97%		100%		100%		100%	99%	NA	
Trichloroethylene	44		14%		97%		98%		99%	79%	NA	
Toluene	2.1		43%		97%		98%		98%	84%	NA	
Xylene	0.21		-86%		95%		100%		95%	52%	NA	
Gross Parameter												
TOC	495		-4%		-41%		-11%			-2%		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)
ND: Not Detected at The Minimum Detection Limits NA : Not Applicable
- Percentage values are cumulative and negative values indicate increasing concentrations
** Supernatant After Coagulation/Sedimentation

physical/chemical treatment processes. This is particularly true for treatment process train 1 (Table 4-9) which produced overall removal rates for measured non-volatiles from the solid phase of 93 to 100% for all cases except Arochlor 1260 which showed only 38% overall removal efficiency for process train 1 and similar results (31%) for process train 2. In general the results obtained for process train 2 for the measured non volatiles were somewhat more variable than for process train 1. This is particularily true for diethyl phthalate (-122%) and acenaphthalene (21%). However, anomalous results were obtained earlier in the process sequence for these two compounds which could account for the comparatively low removal efficiencies observed for these two compounds.

- The measured volatile organics were very effectively removed from the aqueous phase by the combined treatment processes. These compounds are characterized by their low solid/water partititioning coefficients. A comparison of the results in Tables 4-9 and 4-12 illustrates the effectiveness of air stripping for volatiles removal. As indicated in Table 4-12, virtually 100% of the measured volatiles had been removed prior to the chemical oxidation step.
- The results shown in Columns 9 and 10 of Table 4-12 should be comparable since these are essentially the same sample. These results show good agreement for seventeen of the twenty measured organics and reasonably good agreement for

the remaining three (ie. aniline, bis(2-chloroethyl)ether and xylene).

- None of the measured volatiles were detected in the filter cake washwater. This is likely due to the combined effects of the air stripping, vacuum filtration and filter cake air drying steps which preceeded the cake washing step. These processes would be expected to remove essentially all of the measured volatiles from the filter cake. Similarily, only three of the measured non-volatiles were detected in the filter cake washwater. These results suggest that filter cake washing is not a particularily effective means of further reducing the concentrations of non-volatile organics in the filter cake prior to solidification.

Individual Treatment Processes

- The emulsion breaking step had very little effect on the concentrations of the measured non-volatile organics in the aqueous phase and produced variable removals (from less than 10% to 100%) for the measured non-volatiles in the solid phase. These results indicate that the combined effects of heat and gravity sedimentation are an effective means of desorbing organics such as aniline bis(2-chloroethyl)ether, phenol, DDT and lindane from solids present in the waste mixture.
- Emulsion breaking had a variable effect on the concentrations of measured volatile organics in the aqueous phase (ie. less than 15% to 100% removals).

- Chemical oxidation did not appear to have a significant effect on the concentrations of any of the measured organics with the possible exception of 4-bromophenyl phenyl ether, benzene, 1,1-dichloroethylene and methylene chloride for the low level spiked waste.
- As expected, air stripping produced significant additional removals of those measured volatile organics which were still present in the liquid phase after emulsion breaking.
- Air stripping also contributed to the removal of some of the measured non-volatile organics from the solid phase (eg. acenaphthalene and 4-bromophenyl phenyl ether).
- Coagulation/sedimentation appears to improve the removal efficiency of the measured volatile organics in the aqueous phase although there did not appear to be any obvious effect on the non-volatiles in the low level spiked waste.

4.4 Treatment of High-Level Spiked Waste

4.4.1 Treatment Process Train 1

Table 4-13 summarizes the operating conditions and key observations for treatment process train 1 with the high level spiked waste. Tables 4-14 and 4-15 summarize the analytical results for train 1.

4.4.2 Treatment Process Train 2

Table 4-16 summarizes the operating conditions and key observations for treatment process train 2 with the high level spiked

Table 4.13 PROCESS OPERATING CONDITIONS FOR TRAIN 1 - HIGH LEVEL SPIKE

PROCESS	OPERATING CONDITIONS				CHEMICALS	OBSERVATIONS
	PH	E'(mV)	TEMP.(C)	CONTACT TIME		
EMULSION BREAKING			80+5	1 hr. mixing 18 hr. separating		It took 3.5 hr. to heat up to 80 'C Sample volume = 5 L
CHEMICAL OXIDATION First Stage	initial 9.81 final 10.01	initial -50 final 343	20+1	1.5 hr. mixing	611 mL 12% NaOCl	Required 6.5 mL 10% Ca(OH)2 to adjust pH to 8.5. Color change from beige to blue/green to brown.
Second Stage	final 8.0	initial 351 final 602		14 hr. mixing	125 mL 12% NaOCl	Required 26 mL 5N H2SO4 to adjust pH to 8 Observed foaming, an orange foam formed
	initial 7.77 final 8.0	initial 340 final 627		4 hr. mixing	64 mL 12% NaOCl	Required 3.1 mL 10% Ca(OH)2 to adjust pH to 8.0
NEUTRALIZATION/ PRECIPITATION	initial 8.0 final 9.5		20+1	30 min. mixing	53.6 mL 10% wt Ca(OH)2	
COAGULATION			20+1	5 min. mixing 2 hr. settling	20 mg/L 0.5% Purifloc	Supernatant 3L
FILTRATION			20+1	Filtering 9.5 min. Drying = 1.0 min. Washing = 3.0 min. Drying = 2.0 min.	Vacuum ~ 20mmHg	Collected - 160 mL Filtrate - 50 mL wash water Filter Cake - 33.5g - 4 mm thick - 74% moisture

Table 4.14 Analytical Results for Train I-High Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	(mg/L)	Filtrate (mg/L)	wash water (mg/L)	filter cake (ug/g)
Non-volatiles										
Acenaphthalene	ND	750	ND	760	ND	790	ND	ND	ND	400
Aniline	180	390	140	85	84	760	ND	ND	ND	ND
Bis (2-chloroethyl) ether	190	930	200	140	58	ND	160	210	99	13
4-Bromophenyl phenyl ether	ND	7200	ND	7000	0.4	6400	11	0.19	0.67	3000
Diethyl phthalate	0.17	5700	0.8	320	ND	710	ND	ND	ND	990
1,2-Diphenylhydrazine*	ND	1100	ND	950	ND	2300	ND	0.35	0.14	1000
Phenol	44	290	140	300	0.3	43	0.6	0.53	0.37	10
p,p'-DDT	ND	260	ND	262	ND	216	0.36	ND	ND	61.8
Lindane	ND	216	ND	125	ND	76.5	0.025	ND	ND	129
Arochlor 1260	ND	25.9	ND	33.9	ND	27	0.098	ND	ND	7.97
Volatiles										
Acrylonitrile	1300		1.7		19		ND			ND
Benzene	87		3.3		5		ND			41
1,1-Dichloroethylene	170		0.48		0.14		ND			ND
Dibromoethane	11		ND		ND		ND			ND
Methylene Chloride	300		2.6		8.7		0.22			ND
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND			ND
1,1,1-Trichloroethane	46		0.9		1.2		ND			ND
Trichloroethylene	150		13		18		0.59			ND
Toluene	36		2.1		7.9		0.048			16
Xylene	7.1		2.9		9.8		0.021			23
Gross Parameter										
TOC	5544		8773		6930			6897		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix-B)
ND: Not Detected at The Minimum Detection Limits NA : Not Applicable
** Supernatant After Coagulation/Sedimentation

Table 4.15 Treatment Efficiency Results for Train I -High Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid	solid	liquid	solid		Filtrate	wash water	filter cake
Non-volatiles										
Acenaphthalene	ND	750	ND	-1%	ND	-5%	ND	ND	NA	47%
Aniline	180	390	22%	78%	53%	-95%	100%	100%	NA	100%
Bis (2-chloroethyl) ether	190	930	-5%	85%	69%	100%	16%	-11%	NA	99%
4-Bromophenyl phenyl ether	ND	7200	ND	3%	-7000%	11%	-21900%	-280%	NA	58%
Diethyl phthalate	0.17	5700	-371%	94%	100%	88%	100%	100%	NA	83%
1,2-Diphenylhydrazine*	ND	1100	ND	14%	ND	-109%	ND	-600%	NA	9%
Phenol	44	290	-218%	-3%	99%	85%	99%	99%	NA	97%
p,p'-DDT	ND	260	ND	-1%	ND	17%	-35900%	ND	NA	76%
Lindane	ND	216	ND	42%	ND	65%	-2400%	ND	NA	40%
Arochlor 1260	ND	25.9	ND	-31%	ND	-4%	-97%	ND	NA	69%
Volatiles										
Acrylonitrile	1300		100%		99%		100%			
Benzene	87		96%		94%		100%			
1,1-Dichloroethylene	170		100%		100%		100%			
Dibromoethane	11		100%		100%		100%			
Methylene Chloride	300		99%		97%		100%			
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND			
1,1,1-Trichloroethane	46		98%		97%		100%			
Trichloroethylene	150		91%		88%		100%			
Toluene	36		94%		78%		100%			
Xylene	7.1		59%		-38%		100%			
Gross Parameter										
TOC	5544		-58%		-25%			-24%		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)

ND: Not Detected at The Minimum Detection Limits NA : Not Applicable

- Percentage values are cumulative and negative values indicate increasing concentrations

** Supernatant After Coagulation/Sedimentation

Table 4.16 PROCESS OPERATING CONDITIONS FOR TRAIN 2 - HIGH LEVEL SPIKE

PROCESS	OPERATING CONDITIONS				CHEMICALS	OBSERVATIONS
	PH	E'(mV)	TEMP.(C)	CONTACT TIME		
EMULSION BREAKING			80+5	2 hr. mixing 23 hr. separating		It took 3.5 hr. to heat up to 80 'C Sample volume = 5 L
AIR STRIPPING			80+5	2 hr. mixing	Air Flow 2 L/min	It took 3 hr. to cool to ambient temperature
CHEMICAL OXIDATION First Stage	initial 9.99	initial -51.5 final 360	20+1	1 hr. mixing	600 mL 12% NaOCl	Color change from beige to blue/green to brown.
Second Stage	final 8.18	final 608		16 hr. mixing	120 mL 12% NaOCl	Required 5N H2SO4 to adjust pH to 8 Observed foaming, an orange foam formed
	initial 7.95 final 8.05	initial 266.7 final 610.6		1 hr. mixing	64 mL 12% NaOCl	
NEUTRALIZATION/ PRECIPITATION	initial 8.05 final 9.5		20+1	30 min. mixing	52 mL 10% wt Ca(OH)2	
COAGULATION			20+1	5 min. mixing 2 hr. settling	20 mg/L 0.5% Purifloc	Supernatant 3.5 L
FILTRATION			20+1	Filtering 9.5 min. Drying = 1.5 min. Washing = 3.0 min. Drying = 2.0 min.		Collected - 150 mL Filtrate - 50 mL wash water Filter Cake - 51.3g - 4 mm thick - 72% moisture

waste. Tables 4-17 and 4-18 summarize the analytical results for these tests.

4.4.3 Discussion of Results from Treatment of High-Level Spiked Waste

The following observations and conclusions have been drawn from the results of treating the high level spiked waste:

- Similar partitioning behaviour was observed for the measured non-volatiles in the high level spiked waste compared to the low level spiked waste. Only aniline, bis(2-chloroethyl)ether, diethyl phthalate, and phenol showed detectable concentrations in the aqueous phase.

Combined Treatment Processes

- For the treated residues (filter cakes), the overall removal rates were more variable for the high level spiked waste compared to the low level spiked waste. Removal rates for the measured non-volatiles ranged from less than 20% (for 1,2-diphenylhydrazine, see Appendix B) to over 85% for phenol, diethylphthalate and aniline although most of the compounds showed removal rates of 40% or more. Similar results were obtained with both treatment trains. (Tables 4-15 and 4-16)
- The measured volatiles were very effectively removed from the aqueous phase of the high level spiked waste in terms of overall percentage removals. Removal rates in all cases were greater than 95%.

Table 4.17 Analytical Results for Train II- High Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		After Chemical Oxidat		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	liquid (mg/L)	solid (ug/g)	(mg/L)	Filtrate (mg/L)	wash water (mg/L)	filter cake (ug/g)
Non-volatiles												
Acenaphthalene	ND	730	ND	340	ND	570	ND	530	ND	ND	ND	390
Aniline	550	310	170	140	280	220	ND	ND	ND	ND	ND	30
Bis (2-chloroethyl) ether	290	530	220	ND	160	160	92	220	110	110	22	210
4-Bromophenyl phenyl ether	ND	6200	ND	3300	0.32	6200	ND	4100	12	0.25	0.13	5000
Diethyl phthalate	ND	5800	ND	29	ND	33	ND	96	ND	ND	ND	28
1,2-Diphenylhydrazine*	ND	1100	ND	520	1.4	900	ND	1600	0.31	ND	ND	890
Phenol	68	220	130	72	120	240	9.8	13	ND	1.1	0.15	26
p,p'-DDT	ND	28.5	ND	79.5	ND	162	ND	103	0.19	ND	ND	118
Lindane	ND	229	ND	0.29	ND	5.1	ND	6	ND	ND	ND	4.7
Arochlor 1260	ND	38.4	ND	5.3	ND	40	ND	26.9	0.095	ND	ND	21
Volatiles												
Acrylonitrile	1600		37		ND		26		72			
Benzene	84		7.3		1.4		2		0.89			
1,1-Dichloroethylene	110		0.88		ND		0.27		0.065			
Dibromoethane	17		ND		ND		ND		ND			
Methylene Chloride	290		9.3		ND		0.36		0.41			
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND		ND			
1,1,1-Trichloroethane	43		0.84		0.35		0.5		ND			
Trichloroethylene	110		18		10		16		0.63			
Toluene	51		3.7		1.7		2.3		0.81			
Xylene	22		3.3		0.75		0.98		0.6			
Gross Parameter												
TOC	4158		7865		8289		7744			6353		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)
ND: Not Detected at The Minimum Detection Limits NA : Not Applicable
** Supernatant After Coagulation/Sedimentation

Table 4.18 Treatment Efficiency Results for Train II -High Level Spike

Measured organics	Feed		After Emulsion Breaking		After Air Stripping		After Chemical Oxidat		See ** Below	After Filtration		
	liquid (mg/L)	solid (ug/g)	liquid	solid	liquid	solid	liquid	solid		Filtrate	wash water	filter cake
Non-volatiles												
Acenaphthalene	ND	730	ND	53%	ND	22%	ND	27%	ND	ND	NA	47%
Aniline	550	310	69%	55%	49%	29%	100%	100%	100%	100%	NA	90%
Bis (2-chloroethyl) ether	290	530	24%	100%	45%	70%	68%	58%	62%	62%	NA	60%
4-Bromophenyl phenyl ether	ND	6200	ND	47%	-540%	0%	ND	34%	-23900%	-400%	NA	19%
Diethyl phthalate	ND	5800	ND	100%	ND	99%	ND	98%	ND	ND	NA	100%
1,2-Diphenylhydrazine*	ND	1100	ND	53%	-2700%	18%	ND	-45%	-520%	ND	NA	19%
Phenol	68	220	-91%	67%	-76%	-9%	86%	94%	100%	98%	NA	88%
p,p'-DDT	ND	28.5	ND	-179%	ND	-468%	ND	-261%	-18900%	ND	NA	-314%
Lindane	ND	229	ND	100%	ND	98%	ND	97%	ND	ND	NA	98%
Arochlor 1260	ND	38.4	ND	86%	ND	-4%	ND	30%	-3060%	ND	NA	45%
Volatiles												
Acrylonitrile	1600		98%		100%		98%		96%			
Benzene	84		91%		98%		98%		99%			
1,1-Dichloroethylene	110		99%		100%		100%		100%			
Dibromoethane	17		100%		100%		100%		100%			
Methylene Chloride	290		97%		100%		100%		100%			
1,1,2,2-Tetrachloroethane	ND		ND		ND		ND		ND			
1,1,1-Trichloroethane	43		98%		99%		99%		100%			
Trichloroethylene	110		84%		91%		85%		99%			
Toluene	51		93%		97%		95%		98%			
Xylene	22		85%		97%		96%		97%			
Gross Parameter												
TOC	4158		-89%		-99%		-86%			-53%		

Note: * Approximate concentration measured as diphenyldiazene(see section B-2, Appendix B)
ND: Not Detected at The Minimum Detection Limits NA : Not Applicable
- Percentage values are cumulative and negative values indicate increasing concentrations
** Supernatant After Coagulation/Sedimentation

- Bis(2-chloroethyl) ether and, to a lesser extent, phenol tend to be the most potentially problematic compounds in terms of their appearance in the filter cake washwater. This behaviour is expected because both these compounds have comparatively high water solubilities combined with comparatively high sediment/water partition coefficients (see Table C-1, Appendix C).
- The relatively low removal efficiencies which have been observed for the filter cake residues of the high level spiked waste emphasizes the importance of obtaining leach test data on samples of solidified residues as a means of assessing the leachability of the measured organics from samples of solidified residue.

Individual Treatment Processes

- As with the low level spiked waste, emulsion breaking had very little effect on the concentrations of the measured non-volatile organics in the aqueous phase and produced variable removals (less than 10% to 100%) for the measured non-volatiles in the solid phase. Aniline bis(2-chloroethyl)ether, diethyl phthalate and lindane tended to be effectively desorbed by the combined effects of heat and gravity sedimentation.
- As expected, emulsion breaking produced much higher calculated removal efficiencies for the measured volatiles in the high level spiked waste compared to the low level spiked waste.

- With the exception of phenol, chemical oxidation did not appear to have a significant effect on any of the measured organics (volatile or non-volatile).
- As expected, air stripping produced additional removals of those measured volatile organics which were still present in the liquid phase after emulsion breaking.
- Unlike the low level spiked waste, air stripping did not appear to have a noticeable effect on non-volatiles in the solid phase for the high level spiked waste.
- Coagulation/sedimentation appeared to have a negative effect on the removals of some measured non-volatile organics in the aqueous phase. This was particularly true for 4 bromophenyl phenyl ether, DDT, lindane and Arochlor 1260 in the high level spiked waste. It is possible that these organics became desorbed from the solids in the waste mixture during the coagulation step. If this were the case, this process seems to have been reversed to a large extent by the filtration step (ie. organic became readsorbed to the filtered solids).

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APPENDIX A

ANALYTICAL PROTOCOLS FOR ORGANIC COMPOUNDS

A.1 Extraction of Base/Neutral/Acid Extractables

The volume of the sample was measured in a 500 mL separatory funnel. Methylene chloride (10 mL) was used to rinse the cylinder and this was transferred into the funnel together with an additional 100 mL of methylene chloride. The pH of the aqueous portion was adjusted to 12 with 6N KOH and it was spiked with deuterated surrogate standards $^{2}H_{10}$ anthracene, $^{2}H_{12}$ benzo(a)pyrene to monitor recovery in the procedure.

The sample was shaken vigorously for 1 minute and when the phases had separated the methylene chloride extract was drained through a 3.8 cm anhydrous Na_2SO_4 column in an Allihn filter. The aqueous portion was reextracted twice using the same procedure with 75 mL of methylene chloride. After the third extraction, the pH of the sample was adjusted to approximately 2 with 6N H_2SO_4 and was extracted an additional three times with 75 mL of methylene chloride. Methylene chloride (20mL) was used to wash the walls of the Allihn filter and suction was applied to recover all traces of the extract. The extract was then rotary evaporated to approximately 15 mL. and quantitatively transferred to a calibrated centrifuge tube to a final volume of 15.0 mL. for GC/MS analysis. This volume required adjustment depending on the concentration levels in the sample. An aliquot of the sample was used for the clean-up required for the analysis of PCB, DDT, and Lindane. Immediately prior to instrument analysis, the sample was spiked with a deuterated internal standard to compensate for variations in injection volume, instrument conditions etc.

A.2 GC/MS Analysis of Extractables

A Hewlett Packard GC/MSD system was used for gas chromatographic/mass spectrometric analysis. The EPA consent decree base/neutral and acid extractable standards were used to establish instrumental sensitivity and to provide identification criteria for sample analysis. In addition, standards for the additional compounds not included in the EPA 625 method were analysed. The conditions employed were as follows:

Gas Chromatography

Injection Mode	Splitless
Column	30 m DB5 x 0.25 mm ID
Column Flow	He @ 20 cm/sec.

Oven Temperature Profile

80°C - 2 min. —

80°C —> 210°C @ 10°/min.; 210°C —> 290°C @ 16°/min.

hold 10 min.

GC/MS Interface - Direct Couple

Transfer Area - 250°C

Mass Spectrometry

Ionization Mode	Electron Impact
Electron Energy	70 eV
Filament Emission	0.5 A
Electron Multiplier	2000 V
Scan	45-550 a.m.u. in .91 s

A.3 PCB/Organochlorine Analysis

One mL of the extract in iso-octane was placed onto a 1.5 cm ID x 40 cm chromatography column packed with 10 cm of 3% water deactivated silica gel. The centrifuge tube was rinsed several times with 2 mL of hexane. These rinses were also applied to the column. The column was eluted with 60 mL of hexane. This eluant is fraction A. An additional 50 mL of benzene was collected in a separate flask as fraction B. The cleaned up extract was rotary evaporated to 1 mL isooctane, quantitatively transferred to a calibrated centrifuge tube and volume adjusted to 1.0 mL for GC/ECD analysis. A method blank and spike of organochlorines and PCB were also carried out to avoid misidentification and to ensure PCB and organochlorines native to the sample would be recovered through the methodology employed.

A.3.1 Instrumental Conditions - PCB/OC Analysis

A.3.1.1 High Resolution Gas Chromatography

Column	30 M x .25 mm ID DB5;
Injector Temp.	250° C
Carrier	He @ 30 cm/sec.
Injection Mode	Splitless 30 sec.
Split	30 mL/min.
Septum Flush	4 mL/min.
Oven Temp.	80° C - 2 min. — 160° C @ 10° /min.
	— 260° C @ 4° /min, Hold
Detector Temp.	300° C
Detector Makeup	30 mL/min. Argon/Methane 95/5

A.4 Solid Samples

A.4.1 Extractables

The sample of filter cake was weighed, sodium sulphate was added and it was extracted with methylene chloride (130 mL) using polytron homogenization. This was repeated with 2 additional aliquots of solvent. The extracts were dried through a column of Na_2SO_4 and evaporated down to 15 mL with solvent exchange into isooctane. The sample extracts were then analysed as for the aqueous samples.

A.5 Volatiles Analysis in Aqueous Samples

The volatiles were analysed by a headspace technique. An aliquot of the sample was diluted with water to a volume of 35 mL and was placed in a 40 mL septum capped vial. A deuterated surrogate standard of toluene - $^2H_8$ and chlorobenzene - $^2H_5$ was spiked into the sample. The vial was then heated in a water bath at 60-65°C for 15 minutes. A portion of the headspace (200 uL) was injected into the GC/MS system. A standard of EPA volatile organics was analysed in the same manner as the sample.

A.5.1 GC/MS Analysis of Volatiles

A Hewlett Packard MSD was used for the analysis of volatile organics.

The gas chromatographic conditions are listed below:

Column:	30M DB-5 x 0.25 mm ID
Temp. Program:	-10°C for 2 minutes
	-10°C to 120°C at 10°C/min.
	-120°C to 200°C at 20°C/min.

Injector: 150° C

Injection Mode: Splitless, time 0.8 min.

The mass spectrometer conditions used in the analysis are as follows:

Electron Impact mode, scanning 45-300 a.m.u. each second

Electron Energy 70 eV

EM Voltage 2000 eV

Emission Current 0.5 A

The U.S. EPA standard mixture prepared by Radian Corporation was used for quantification and confirmation of the presence of volatile organics. The GC/MS was calibrated with PFTBA (FC-43).

Quantification was performed using the external standard method, as detailed in the Federal Register, on peak areas from reconstructed ion plots for the quantification ions listed in the EPA Federal Register. Assurance of correct identification was performed using the secondary ions and in the case of higher level determinations, using the full scan spectra.

A.6 Volatile Analyses in Methanol Samples

A portion of the methanol was diluted with water to an appropriate concentration level. This was then treated in the same manner as for the aqueous samples.

APPENDIX B

DISCUSSION OF ANALYTICAL PROCEDURE AND RESULTS

B.1 Analysis of Volatiles

The analysis of cryotraps posed some analytical problems since so much material was present in them. Initial attempts were made to transfer the contents of the trap to a tenax trap by warming the cryotrap in a sealed system where the volatiles would be passed through tenax. Adsorption would occur followed by a transfer of the adsorbed volatiles to a GC/MS system using a thermal desorption technique. This approach saturated the GC/MS system, both column and pre-amplifier, and so only estimated values are available for the two samples treated this way. (464729 - Emulsion breaking trial run and 864730 Air stripping trial run).

A different approach was therefore taken for the other cryotraps. The cryotrap samples which had been transferred to the tenax traps were then thermally desorbed into 35 mL of methanol for a period of 15 minutes. A portion of the methanol was then analysed by the headspace technique used for the other aqueous/liquid samples.

The high levels of organics in the cryotraps were likely to saturate the tenax capacity for adsorption and a further modification of the analytical approach was necessary. Three of the traps were not transferred to tenax as an initial step and these were stored in the freezer until analysis. A known portion of methanol, sufficient to almost fill the cryotrap, was added to the sample and mixed. The final volume was then measured and a portion of the methanol subjected to GC/MS analysis by the headspace technique.

The latter method is expected to provide the most accurate data. The method involving adsorption onto tenax prior to analysis suffers from the possibility of break-through in the tenax and

subsequent losses of analytes. These should be considered as lowest possible numbers. The two samples thermally desorbed directly into the GC/MS will have concentrations underestimated because of tenax breakthrough but also because of GC and MS overloading.

The analysis of 1,1,2,2 tetrachloroethane was complicated by its low vapour pressure and its poor mass spectrometric response compared to some of the other compounds such as benzene and toluene. The analysis of dibromomethane suffered from similar constraints but, in addition, was at a concentration level ten times lower than other components.

The range of mass spectrometric response in the volatiles analysis and the volatility range created analytical problems in selecting a dilution factor which would prevent the high response compounds such as benzene from saturating the mass spectrometer while enabling the analysis of compounds such as tetrachloroethane and dibromoethane.

B.2 Analysis of Extractables

The analysis of 1,2-diphenylhydrazine was complicated by its conversion within the spiking matrix to diphenyldiazene. The latter compound has a very different mass spectrum from its parent compound. No sign of the 1,2-diphenylhydrazine was apparent when the spiking solution was analysed in conjunction with the samples. As a result, the samples were analysed for diphenyldiazene instead of 1,2 diphenylhydrazine but, since a standard of the former was unavailable, an estimate is made of the concentration level.

Spiking levels of deuterated analogues were evaluated based on a "best guess" of the final volume of extract required for the GC/MS analysis. This volume was subject to change depending on the sample. The greater dilution, the larger the variation to be expected in the surrogate recoveries since the analytical conditions were different from the design conditions. The recoveries are therefore not altogether indicative of the recoveries of native contaminants.

APPENDIX C

PROPERTIES OF CHEMICALS

Table C-1 Properties of Chemicals

Specified Organics	Molecular Weight	Boiling Point (C)	Vapour Pressure (torr)	Water Solubility (mg/L)	Octanol/Water Partition Coefficient	Sediment/Water Partition Coefficient
acenaphthene	154.2	279	1.55 x 10E-3 (25 C)	3.42 (25 C)	9.6 x 10E3	4.6 x 10E3
aniline	93.13	184		34000		
bis (2-chloroethyl) ether	143	178	0.71 (20 C)	1.02 x 10E4	29	13.9
4-bromophenyl phenyl ether	249.11	310.14	1.5 x 10E-3 (20 C)	4.8 (25 C)	8.7 x 10E4	4.2 x 10E4
diethyl phthalate	222.2	298	3.5 x 10E-3 (25 C)	896 (25 C)	295	142
1,2-diphenylhydrazine	184.2		2.6 x 10E-5 (25 C)	1.84 x 10E3	871	418
phenol	94.11	181.75	0.341 (25 C)	9.3 x 10E4 (25 C)	30	14.2
p,p - DDT	354.5	185	1.9 x 10E-7 (25 C)	5.5 x 10E-3 (25 C)	8.1 x 10E6	3.9 x 10E6
lindane	291	-	1.6 x 10E-4 (20 C)	7.8 (25 C)	7.8 x 10E3	3.8 x 10E3
Arochlor 1260	375.7		4.05 x 10E-5 (25 C)	2.7 x 10E-3 (25 C)	1.4 x 10E7	6.7 x 10E6
acrylonitrile	53.1	78.5	100 (22.8 C)	7.9 x 10E4 (25 C)	1.78	0.85
benzene	78.12	80.1	95.2 (25 C)	1780 (25 C)	135	65
1,1-dichloroethylene	96.94	37	591(25 C)	400(20 C)	135	65
dibromoethane	187.8			4.3		
methylene chloride	84.8	40	3.76 x 10E3 (20 C)	6.45 x 10E3 (20 C)	8.9	4.3
1,1,2,2-tetrachloroethane	167.85	146.2	5 (20 C)	2.9 x 10E3 (20 C)	245	118
1,1,1-trichloroethane	133.41	74.1	123 (25 C)	720 (25 C)	320	152
trichloroethylene	131.39	87	57.9 (20 C)	1.1 x 10E3 (20 C)	263	126
toluene	92.13	110.6	28.7 (20 C)	534.8	620	300
xylene	106.16			175		

Table C-2 Typical Minimum Detection Limits of Organic Compounds

Specified Organics	Minimum Detection Limits	
	liquid mg/l	solid ug/g
acenaphthene	0.05	1
aniline	0.05	1
bis (2-chloroethyl) ether	0.05	1
4-bromophenyl phenyl ether	0.05	1
Diethyl phthalate	0.05	1
1,2-diphenylhydrazine	0.05	1
phenol	0.05	1
p,p - DDT	0.001	0.05
lindane	0.001	0.05
Arochlor 1260	0.003	0.15
acrylonitrile	0.5	50
benzene	0.02	4
1,1-dichloroethylene	0.05	10
dibromoethane	0.2	20
methylene chloride	0.05	10
1,1,2,2-tetrachloroethane	0.05	10
1,1,1-trichloroethane	0.05	10
trichloroethylene	0.05	10
toluene	0.02	4
xylene	0.02	4